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Protocol 33 and Beyond

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ABSTRACT

In its first use in simulating engine operating conditions, the Thermo-oxidation Engine Oil Simulation Test (TEOST*) was applied to the study and deposition measurement of engine oils under turbocharger operating conditions in a technique called Protocol 33C. Building on a previous paper, the authors present data and discussion on the factors involved in the high temperature cyclic test conditions of the TEOST and reemphasize differences between it and steady-state oxidation tests.

Some new studies are discussed which involve application of the TEOST apparatus at lower temperatures using simulated crankcase gases. These are believed more reflective of piston deposit conditions. Preliminary studies indicate that results from this new lower-temperature protocol correspond to known reference engine oils in regard to piston deposits and other measured factors.

KEY WORDS

Oxidation, cyclic oxidation tests, TEOST, engine oil, turbocharger, engine oil deposits.

INTRODUCTION

A previous paper [1] presented the concept of the Thermo-oxidation Engine Oil Simulation Test (TEOST) apparatus shown in **Figures 1a** (the unit) and **1b** (the Depositor Rod and casing zone). Although the instrument was developed to study engine oils and their response to conditions simulating those in the turbocharger, it was anticipated that this instrument would in time provide a variable test bed for other studies of the thermal and oxidative properties of engine lubricants.

Among the variables designed into the instrument, the TEOST included different zones for engine oil conditioning (Reactor) and stressing (Depositor), variable temperatures in each, programmable cyclic stress temperatures, variable flow rate and volume of lubricant, various means of introducing liquids and gases to either the conditioning or stressing zones, and a variable length of test time.
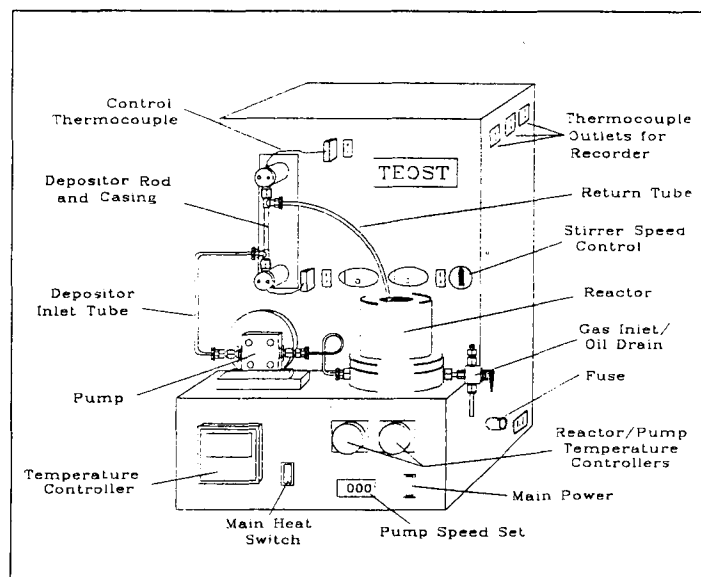
* TEOST is a registered trademark of Tannas Co.

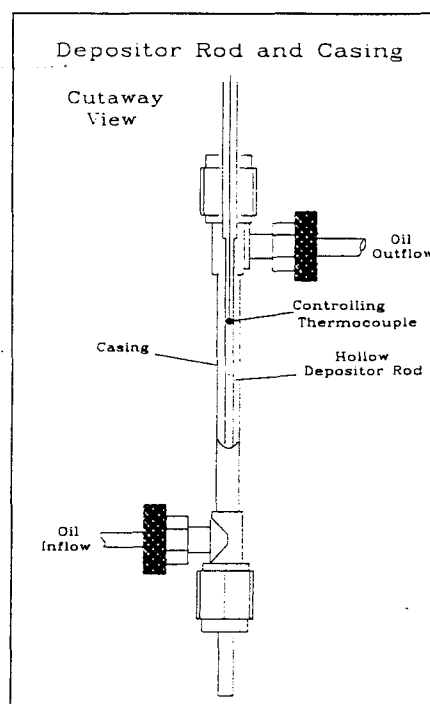**Figure 1a:** Sketch of the TEOST apparatus

Figure 1b: Sketch of the Depositor Rod and casing.

In addition, it was possible to add reactants to the charge of the engine oil tested to encourage response or to further stress the oil under the test conditions. The availability of several thermocouple plugs on the apparatus permitted temperature control and/or measurement at a number of locations in the oil stream.

With so many variables, considerable developmental effort was required to find those operating conditions giving results corresponding to the four turbocharger reference oils available at the time. These operating conditions were identified as Protocol 33C and involved a cyclic high-temperature heating program. Results shown in the first paper are replotted in **Figure 2**.
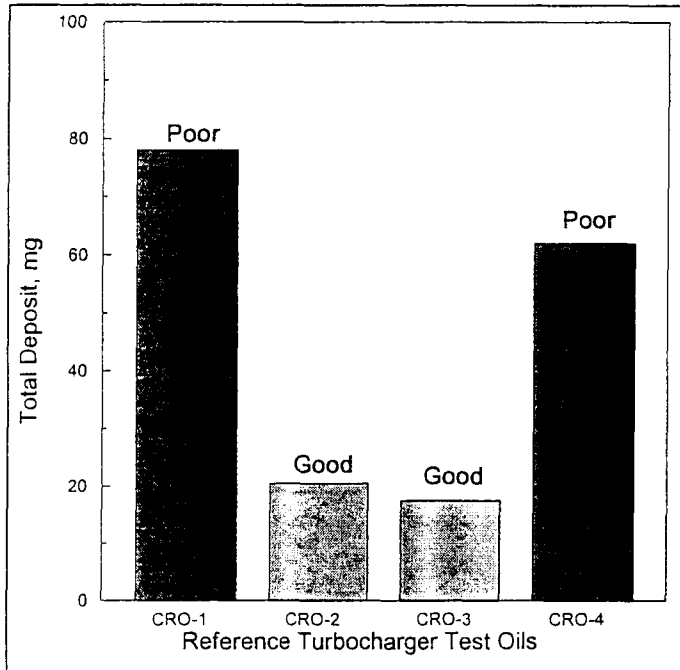

Figure 2: Correlation of TEOST Protocol 33C with field data.

This paper presents further background on the information gathered using Protocol 33C and some preliminary information on the development of another protocol for lower temperature, more oxidation-rich crankcase operating conditions.

PROTOCOL 33C

To understand the background and rationale of Protocol 33 (developed on the prototype), and 33C (using the commercial test apparatus), it is important to understand the turbocharger conditions that are being simulated. For reference, **Figure 3** shows a cutaway sketch of a turbocharger housing.

Previous work by other investigators [2-8] using reference oils of known turbocharger performance showed, among other factors, that deposits were influenced by cyclic conditions, very high temperatures, high NO_x levels, as well as dispersant additive concentration and effectiveness. These parameters were considered in the development of Protocol 33C. Two of the reference oils used in developing Protocol 33C had been previously used in European turbocharger test studies [2].
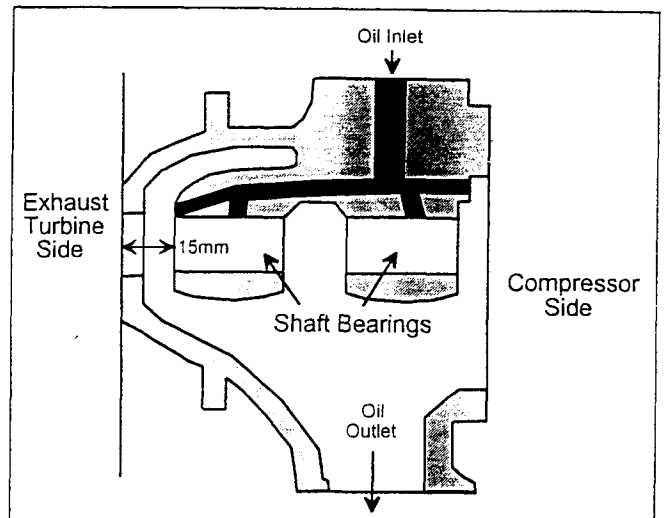

Figure 3: Cutaway sketch of a turbocharger. (Adapted from Reference 2.)

Since engine oil is supplied copiously to the turbocharger as both a coolant and a lubricant during operation of the engine, earliest stages of turbocharger coking occur just after the engine is shut off. Once the engine is stopped, so too (in most designs), is engine oil circulation. Engine oil which on flowing past the hot turbine shaft cooled it, now is forced to sit quiescently in full contact with a growingly hot shaft reflecting the 600° or 700°C temperature of the turbine wheel extending into the exhaust stream.

As shown in **Figure 3**, this turbine wheel is only a few millimeters away from the now quiescent engine oil in the galleries used to bring the flowing oil in contact with the shaft-bearing area. Temperatures well in excess of 400°C have been recorded [2] in this zone, once the engine was shut down.

It is believed that, since limited quantities of oxidizing species are likely available, the hot steel thermally decomposes the oil and carbonaceous deposit formation is enhanced causing the galleries to become progressively plugged. This, in turn, slows the rate of oil circulation during operation and increases further growth of the carbonaceous mass. Ultimately, oil flow stops, denying both lubrication and cooling to the turbocharger shaft and the turbocharger fails.

Thus, in setting up the laboratory test, there were two factors considered critical to the TEOST turbocharger deposit simulating protocol:

1. A cyclic high temperature environment

2. An environment somewhat biased toward thermal rather than oxidative deposition.

These conditions could, of course, be met by a number of possible test protocols. Fortunately, the two reference oils from North America and the two from Europe were available around which to investigate the appropriate balance of test conditions to rank these oils properly. Protocol 33C was not an arbitrary choice of operating parameters as a number of protocols were studied before the final test conditions were chosen (although other or better ones are certainly possible).

As was detailed by the authors in the previous paper on the TEOST [1], the cyclic temperature control in Protocol 33C is as indicated in **Table 1**. Other requirements of the test are

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given in **Table 2** including quantity of test oil, flow rate through the Depositor, use of a small amount of an oil-soluble iron compound (to emulate the role of iron in the engine environment as a catalyst), and the injection of moist air and N_2O, etc.

Summary of Protocol 33C - Although the reader is directed to the authors' first paper [1] for more detailed information, a quick summary of Protocol 33C may be helpful.

The Depositor Rod is cleaned and weighed to a tenth of a milligram after which it is carefully assembled within the Depositor Rod casing (see **Figure 1b**). The test is then run under the conditions of **Tables 1 and 2** following which the Depositor Rod is carefully removed from the casing, washed and weighed. Similarly, at the conclusion of the test, all of the test oil is filtered and the particles obtained from this filtration weighed to a tenth of a milligram. These two weights combined constitute the weight of deposits generated by the engine oil.

From the operational viewpoint, the test begins by quickly bringing the engine oil to 200°C (from room temperature) at which point the first cycle is run. Eleven more cycles follow and the test is concluded after which the weighings are completed. A strip chart record of the run provides pertinent information as a thermogram of the response of the Depositor Rod temperature to those factors influencing it.

DEPOSIT FORMATION

TEOST AND FIELD DEPOSIT COMPARISON - As mentioned earlier, the cyclic character of deposit formation at high temperatures in the turbocharger as a result of periodic engine shutdown [2] was emulated by the cyclic nature of Protocol 33C but in a much more rapid time frame.

In order to compare the characteristics of the turbocharger and Protocol 33C deposits, x-ray fluorescence spectra of deposits taken from field-failing turbochargers were obtained. A JVAR EX-6000 Series, ED-XRF spectrometer was used for this work.

Figures 4a through **4d** are the spectra of four field samples of carbonaceous deposits taken from field-failed turbochargers. Mileage on the turbochargers before breakdown and repair ranged roughly from 14,000 to 50,000 miles. While the oils that had been used in these turbochargers and their

Figure 4a-d: Shown below and on the following page are X-ray fluorescence spectra of four deposits from four field-failed turbochargers. These spectra were obtained by direct bombardment of the samples. Peaks reflecting the x-ray source are shown by asterisks.

Table 1 - Program Steps, Protocol 33C

Program Step	Hold	Ramp	Time, min.	Temp. °C
0*		✓	immed	RT-200
1	✓		1.25	200
2		✓	1.00	200-480
3	✓		2.00	480
4		✓	4.00	480-200
5	✓		1.25	200
			Total: 9.50	

*Cycle 1 only; Cycles 2 to 12: Program Steps 1 to 5

Table 2 - Operating Parameters

Parameter	Value
Total oil volume	116 mL
Oil pumping rate	0.40 g/min
Reactor temperature	100°C
Moist air flow	3.6 mL/min
N_2O flow	3.6 mL/min
Iron naphthenate	100 PPM Fe
Position of controlling thermocouple in Rod	~71 mm from top of Rod

Mid-West
49,500 miles

except by being incorporated in the deposit from the engine oil. Among the spectra, calcium, zinc, and copper represent additive elements -- particularly zinc. Iron is presumed representative of wear while copper could be representative of either additive or wear.

In comparison, **Figures 5a** and **5b** are scrapings of carbonaceous material from TEOST Depositor Rods. **Figure 5a** is from multiple Rods run on the same borderline depositing reference -- an API SF, SAE 10W-40 engine oil, Reference Engine Oil B -- while **Figure 5b** is obtained from this same oil using a 24-cycle run to enhance the quantity of deposit. A serrated plastic spatula was used in scraping to avoid removing any iron from the Rod. The spectra are quite similar and show calcium and zinc as well as the iron which was added to the oil initially in the form of 100 PPM iron naphthenate.
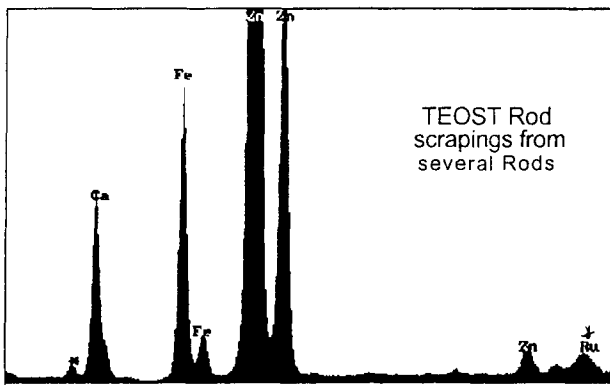

TEOST Rod scrapings from several Rods

Figure 5a: X-ray fluorescence spectra of deposits from several TEOST Rods obtained in tests using Reference Engine Oil B.

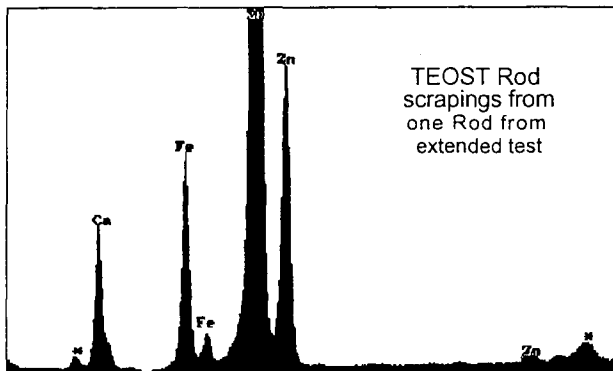TEOST Rod scrapings from one Rod from extended test

Figure 5b: X-ray fluorescence spectra of deposits on the Rod of one extended TEOST test Rods using Reference Engine Oil B.

Despite the fact that the field samples and the TEOST deposits are not necessarily of the same oils with the same concentrations of the same additives, the conclusion is strong -- both the turbocharger and the TEOST create similar deposits from both the surface-active and heat-reactive nature of the additives and the oil. Moreover, considering the surface-active and heat-reactive nature of some of the additives used in formulating oils, these additives would be expected to be the first molecules to experience the heat moving down the turbine shaft. It is thus reasonable that some of the first deposits will be of these thermally degraded additives and that the process of building up deposits would continue to include such materials as part of the oil composition.

Influence of Additives on Turbocharger Deposits - From another viewpoint, it is interesting to speculate on what would happen to simple base oils in the turbocharger environment previously described. In the absence of additives, it would seem likely that the oils might simply volatilize leaving little or no deposits. Thus, although the carbonizing of the oil is a part of forming turbocharger deposits, the additive type and concentration may provide the matrix for turbocharger deposits and their growth.

On the other hand, some additives such as certain detergents seem to work to prevent carbonaceous buildup in the turbocharger [2]. These may work by removing decomposition products before adherence becomes a flow-reducing problem.

Overall, it seems that additives both participate in and work against carbonaceous build-up in the turbocharger and perhaps in other hot areas of the engine. This suggests a careful balance of additive type and content is needed for the lubricant service intended. As is often the case. 'more' is not necessarily synonymous with 'better'.

DEPOSITOR EFFECTS ON THE OIL

GENERAL - The process of producing deposits on the Rod infers a process affecting the oil being tested as well. Thus, if additives are being decomposed on the Rod, the oil should show changes in the additive content and chemistry. Similarly, if the conditions of deposit formation are in an environment somewhat limited regarding oxidizing sources then the oil should reflect changes that exclude much oxidation or evidence of oxygen uptake. Rather, oxygen depletion might be more the case.

FOURIER TRANSFORM INFRARED STUDIES - In an effort to look more carefully at the engine oil decomposition process going on in the Depositor and recognizing that simply collecting the final oil sample was not sufficiently informational, another sampling approach was used. In this approach, the previously mentioned extended Protocol 33C was run for 24 cycles on a reference oil which normally would produce about 60 mg of total deposits. One of the more obvious results of such an extended run was the increased deposit level subsequently used to obtain the x-ray fluorescence spectra shown in **Figure 5b**. Total deposits were 165 mg of which 159 mg were on the Depositor Rod.

During this run, the output of the Depositor casing return tube was sampled every two minutes during the 3rd, 14th, and 22nd cycles. This procedure was followed to permit both determination of transitions in composition during a cycle as well as any longer term, progressive transition during the 24-cycle run. At the flow rate used in Protocol 33C, the amount of oil passing through the Depositor Rod casing in 12 cycles is about 54 mL. With a total oil charge of 116 mL this is somewhat less than half the amount of oil available in the Reactor. However, in the design of the TEOST, the oil that has passed through the Depositor is stirred into the oil remaining in the Reactor. Consequently, the oil flowing to the Depositor from the Reactor gradually carries more and more of the Depositor degraded molecules as well as having endured continuous exposure to the conditions in the Reactor.

In the case of the 24-cycle extended test on Reference Engine Oil B, virtually all of the oil has passed through the Depositor zone. (Even though the oil is re-circulated, vertical re-mixing is limited and the Reactor continues to feed the fresh oil at the bottom of the Reactor to the pump and Depositor Rod.)

Fluid samples collected during the selected cycles were then analyzed using the transmission mode of a Nicolet Model 8210E FTIR. Each sample from each cycle was analyzed. FTIR analysis of the fresh Reference Engine Oil B is shown in **Figure 6** for the reader's reference.
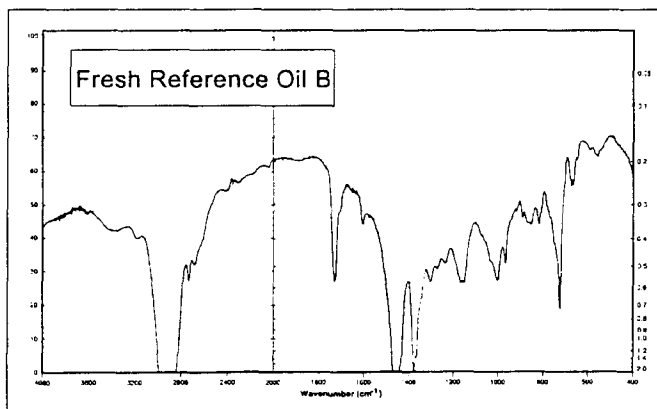

Figure 6: FTIR spectra of fresh oil used in study.

Similar FTIR analyses were made of all the samples collected during the 24-cycle extended TEOST 33C analysis of Reference Engine Oil B. Then, using FTIR subtraction techniques for these spectra, the spectrum of the fresh oil (see **Figure 6**) was subtracted from the spectrum of each of the samples and these results 'stacked' above one another to make reading the transition occurring with passage through the Depositor Rod casing easier to see. (Occasionally, during higher humidity atmospheric conditions, the spectra showed small closely spaced peaks that were aberrations in the spectra and not associated with them.)

Figures 7a-c show these subtracted spectra. Both increasing and decreasing peaks are noted in the subtractive spectra. Increasing peaks are evidence of increasing presence of the molecules causing the peak and *vice versa*. It should be kept in mind that the oil first flowing from the Depositor Rod casing return tube at the beginning of a cycle is the remnant of
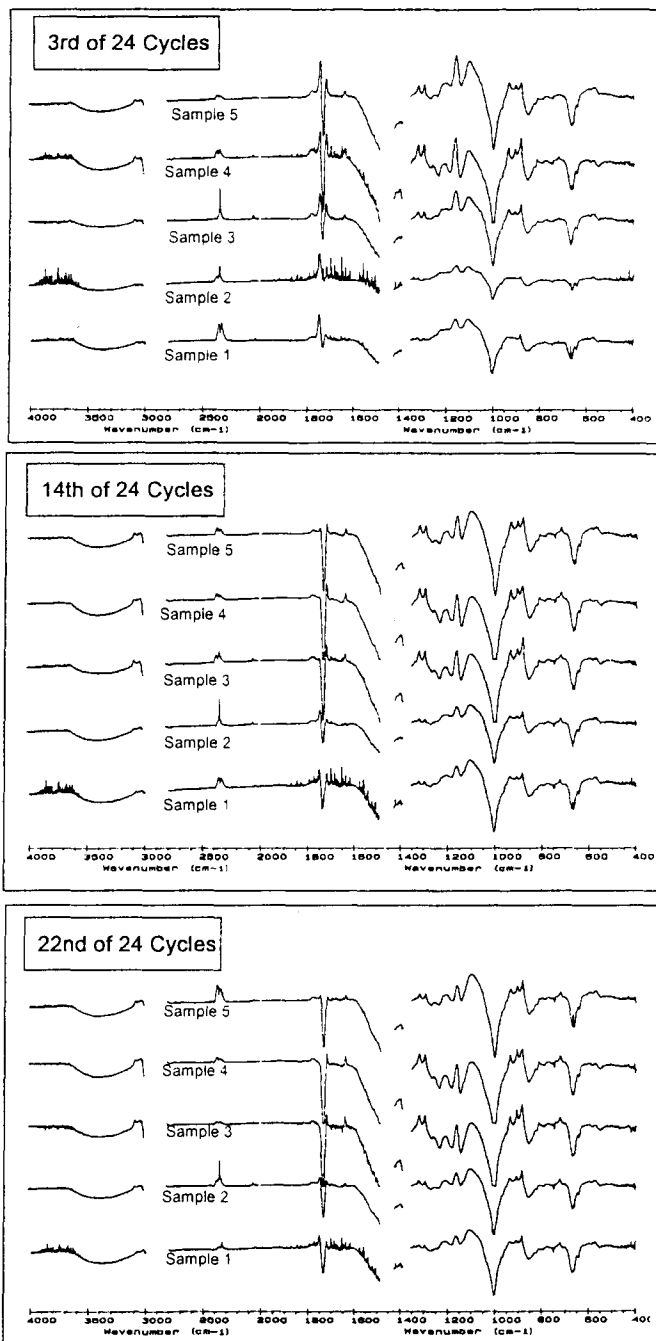
Figure 7a-c: FTIR subtraction spectra of two-minute samples of Reference Oil B at selected cycles in extended TEOST Protocol 33C study.

the previous cycle just coming through the return tube. From this point of view, the samples are related to the program as shown in **Figure 8** on the following page.

If the spectra from Cycle 3 are analyzed, evident changes are shown in certain sections. For example, generally the spectra show considerable changes in the 960 to 1020 cm^{-1} region associated with the additive components. Not surprisingly at this early stage of the TEOST test, there is also evidence of limited oxidation and nitration in the regions of 1670 to 1800 cm^{-1} and 1600 to 1650 cm^{-1}, respectively. Sulfonation is also observed in the 1120 to 1180 cm^{-1} region which can also include the spectra of sulfonate detergents.

Figure 8: Association of the five sampling periods with the TEOST 33C program. Only the first two cycles are shown.

More specifically, from the spectra of Sample 2, some decomposition of the additive is already shown and is most likely evidence of the precursor effects of time spent in the 100°C Reactor. As the cycle proceeds from Sample 3 to 4 to 5 and back to Sample 1, the additive components show considerable decrease as the oil is exposed to the very hot zone of the Rod. Simultaneously, there is a marked decrease in some of 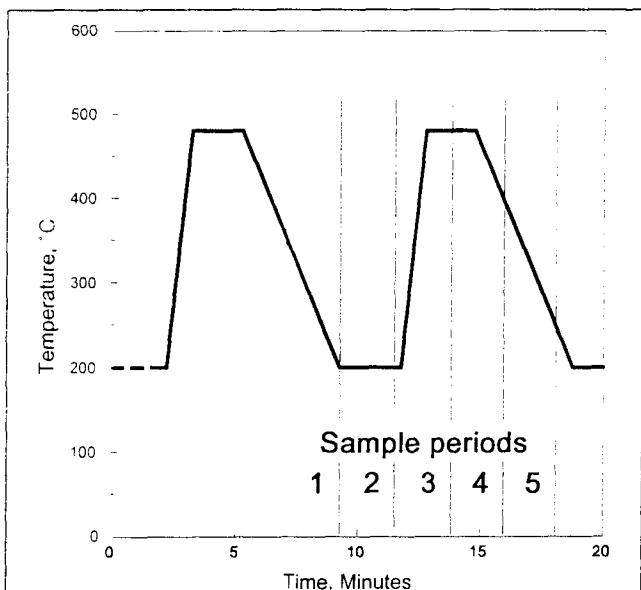the oxygenated species at about 1730 cm^{-1} and some increase in other oxygenated species on either side of this value -- at about 1710 and 1750 cm^{-1}. Sulfonation becomes very evident in the process, perhaps as a product of the decomposition of dithiophosphates. Some slight nitration can be observed.

At Cycle 14, additive decomposition shown by Sample 2 is considerably greater than that of Sample 2 for Cycle 3. Further damage is done during Cycle 14 but in general much of the additive has been affected already. Loss of oxygenated species continues including loss of oxygenated species produced in Cycle 3 indicating the competition for oxygen during the thermal formation of carbonaceous material from hydrocarbons. Sulfonation continues to remain relatively high and nitration is somewhat more evident early in the cycles. At Cycle 22, additive decomposition levels are essentially unchanged from Cycle 14 as are the levels of sulfonation and loss of oxygenated species. Nitration increases slightly.

While there is much more information to be gleaned from these FTIR data and other such experiments, the present results set a scenario of carbonaceous deposit formation at very high temperatures and relatively low, and progressively lower, levels of oxygen-containing species -- a condition not unlike that believed operative in the turbocharger after shutdown.

ENGINE OIL HEAT TRANSFER EFFECTS

GENERAL - It is a matter of both interest and understanding to look more deeply into information which can be gathered in running TEOST Protocol 33C. One of the better tools for letting the instrument 'express itself' is the strip-chart recorder often used with the instrument to produce a thermogram of the analysis. Another source of information is the various sampling processes which can be practiced using the oil in the Reactor or the oil coming from the Depositor.

HEAT TRANSFER ROLE OF THE ENGINE OIL - Engine oil is an important source of heat transfer in the engine and is so used in conjunction with its lubrication role in the turbocharger. However, heat transfer from a metal surface to the engine oil is strongly conditioned by several physical and chemical factors and these factors become operative or more evident when using a cyclic heating technique such as TEOST Protocol 33C. Moreover, these physical and chemical factors would be expected to be closely associated with the engine oil formulation.

Factors Affecting Heat Transfer - The relationship affecting the flowing oil and the Depositor Rod surface is influenced by at least seven factors associated with the oil tested. Specifically, the oil and Rod affect heat transfer from the Rod surface by

1. the rate of heating the Rod surface,

2. the presence and effect of any layer of surface-active additives in the oil,

3. the heat conductivity of the bulk oil,

4. the rate of oil flow,

5. the heat capacity of the oil,

and, as the test progresses,

6. the rate and kind of deposit formation, and

7. the effects of changes in the oil during the test on the previous five factors.

In Protocol 33C only Factors 1, 2, 6, and 7 are considered significantly variable, and, as will be discussed, Factor 1 -- though set during calibration of the TEOST apparatus -- is significantly influenced by Factor 2. Factor 2 is well known to be highly influential among all other heat transfer factors [9].

Evidence of the initial influence of Oil Additives on Heat Transfer - To determine if the effect of Factor 2 during the initial test cycles varies from engine oil formulation to formulation in Protocol 33C, support would be generated by proper response to two tests. That is,

1. repeat tests on the same oil would give the same cyclic response in the thermogram generated by the strip chart as well as in the deposits generated by the test, and

2. tests on oils of different formulations would give different responses to the cyclic conditions although not necessarily different levels of deposits.

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Figures 9a and b are two of a number of analyses of a Reference Engine Oil A, an oil having moderate deposit-forming tendency. The thermograms show that the first condition is met (within the limitations of the method) both in the form of a certain appearance of the cyclic variation during the initial two or three cycles and in the resulting deposits.
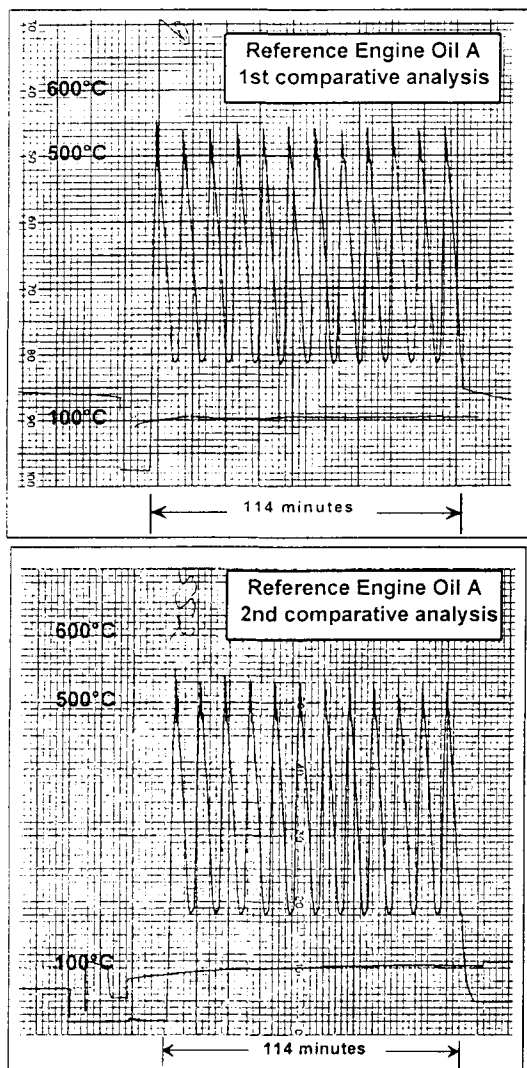
Figures 9a-b: Comparative TEOST Protocol 33C thermograms of two analyses of Reference Engine Oil A. Total weights were 30.5 and 29.1 mg, respectively.

Similarly, **Figures 10a** and b are two analyses of Reference Engine Oil B known to contain a different additive system and to have a borderline level of deposit formation. These latter oils demonstrate a different form of cyclic response to Protocol 33C. It is evident that the two analyses are again quite similar to one another in cyclic form -- and somewhat in the deposit level obtained -- but significantly different from the form and values of Engine Oil A. (Interestingly, in the case of Engine Oil B the comparatively higher deposit level corresponds to the reduced number of cycles before the thermogram shows significant loss of heat transfer.)

Many tests conducted by the authors on a number of different additive formulations have shown such differences or similarities depending on the additive chemistries involved. Obviously, the cyclic form and deposit level for a given oil will be influenced by test to test variation, but the similarities

are evident. In fact, occasionally the cyclic response shown on the strip chart has been used to check that the appropriate oil has been analyzed.
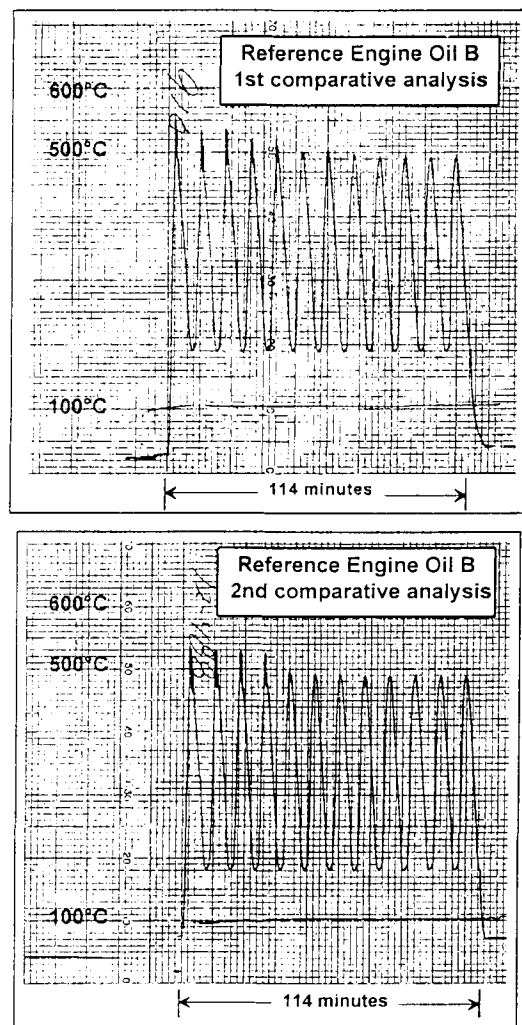

Figure 10a-b: TEOST Protocol 33C thermograms of first and second comparative analyses of Reference Engine Oil B. Total weights were 55.3 and 73.2 mg, respectively.

Heat Transfer and Deposit Formation - The relationship among cooling rates, oil and additive decomposition, and deposit formation is at the heart of turbocharger deposits and malfunction. Similarly, the evidence given by the TEOST Protocol 33C that different engine oils show different heat transfer rates depending on their composition, would also be expected to be associated with deposit formation during the later cycles of this 12-cycle test.

Deposits would, first of all, be expected to occur in the hottest zone of the Depositor Rod which is chosen as the position of the controlling/sensing thermocouple. Secondly, it would be expected that any significant deposit would affect heat transfer from the Rod to the oil. Thirdly, it would be expected that the curtailment (or enhancement) of heat transfer through the deposit to the oil would curtail (or increase) the call for heat by the thermocouple controlling energy to the Rod. Consequently, the thermogram produced by the strip-chart recorder should be capable of being interpreted regarding both heat transfer and deposit formation.

Cyclic Programming and Cyclic Response - In view of the foregoing considerations concerning heat transfer, it is not expected -- or desired -- that each oil would exactly parrot the cyclic temperature ramps and holds of **Table 1** programmed into Protocol 33C. Rather, to indicate its expected turbocharger performance, each engine oil must be free to respond to the TEOST program with whatever heat-transferring and deposit-resisting characteristics it may have.

Considering the nature of the thermoelectronic controls of the TEOST, wherein band width, reset, and rate derivative values are carefully set and calibrated, study of the strip-chart thermograms is instructive. Cyclic response to heat transmission from the Rod to the oil through the surface adsorbents -- additives and/or deposits -- produces characteristic thermographic patterns.

If, for example, the test oil is highly effective in absorbing energy from the Rod, the controlling/sensing thermocouple will provide more heat to the Rod to compensate. Under these conditions, it is likely that after the ramp of Program Step 2 in **Table 1**, the temperature indicated by the controlling/sensing thermocouple will show an excursion peak beyond the 480°C upper limit of Steps 2 and 3. Moreover, since the cooling rate will also be rapid, there may be second or third excursion peaks during the two-minute hold period of Step 3. Such oil thermograms have already been shown in the analyses of Reference Engine Oil A in **Figures 9a** and **b**.

On the other hand, if the test oil is not highly effective in absorbing energy from the Rod, the rapidly accumulating Rod heat will influence the controlling/sensing thermocouple to shut down the rate of heat production. Consequently, the thermocouple will more effectively anticipate the 480°C limit and the system will tend to have little if any excursion and only one peak will be produced.

Single cyclic peaks have frequently been associated with higher deposit levels. Such deposits often seem to act as insulators of heat transfer to the oil and consequently modulate the heat rise as the controlling thermocouple senses adequate levels and rates of heating the Rod. The consequence is, again, a single peak during the thermographic cycle after the ramp of Program Step 2 and during the two-minute hold of Program Step 3. (However, very infrequently -- and seemingly associated with particular chemistry -- deposits comparatively effective in heat transfer have been observed.)

It is speculated that when the deposits begin to accumulate on the Rod during test of an oil initially effective in absorbing energy from the Rod, there will be a transition from temperature excursion peak(s) to the non-excursion single-peak deposit response at the point in the cycles where the deposits are sufficiently effective in insulating the Rod. This behavior is evident in the thermograms shown in **Figures 10a** and **b** for Reference Engine Oil B.

In some engine oil formulations, deposited carbonaceous material will spall off the Rod and the Rod will continue to show its original heat transmission characteristics.

Base Oils, Heat Transmission, and Deposit Formation - In the absence of additives and in an environment of relatively low oxidative stress but high temperatures, base oils give an interesting response to the TEOST Protocol 33C. As might be expected, without the additive to form a deposit matrix and at temperatures high enough during the heat ramp to volatilize some of the oil in immediate contact with the Rod, few deposits are formed. Particularly, at lower viscosities and higher volatilities, a gaseous Rod/oil interface would develop during the high heat portion of the cycle, and very little deposit would be expected on the Rod or in the oil.

Some TEOST Protocol 33C experiments were run on both a 92 Viscosity Index (VI) paraffinic Solvent Neutral and 53 and 54 VI naphthenic base stocks, all simple oils without additives.

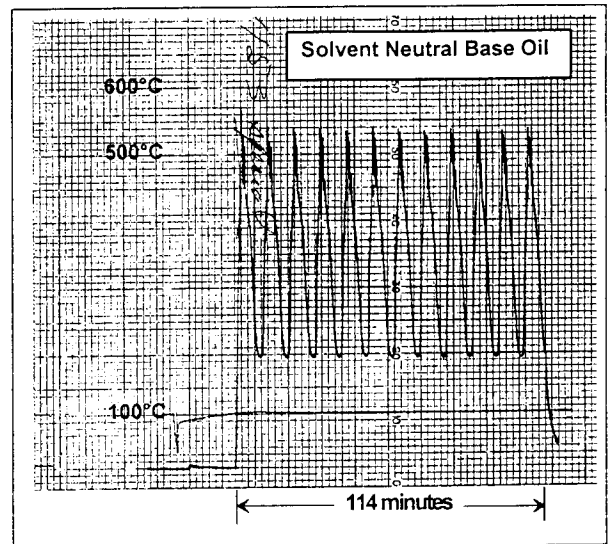The thermogram of the lower viscosity, paraffinic base oil

Figure 11: TEOST Protocol 33C thermograms of a simple 100 Solvent Neutral base oil. Total weight was 6.2 mg.

is shown in **Figure 11**.

Interestingly, on the basis of the thermogram, this oil seems to behave as a good heat transfer oil. Since the thermal conductivity of most base oils are the same, most likely the heat handling response of this oil reflects the greater mobility of its lower molecular weight components. Lower molecular weight components could tend to more readily volatilize and form a gaseous zone around the heated rod. In any case, little deposit is shown.

In comparison, two naphthenic base oils were analyzed using Protocol 33C. Viscosities of these base oils were considerably higher than that of the paraffinic base oil. That is, the former was about 4 cSt at 100°C while the naphthenic base oils were 9.2 and 10.4 cSt at 100°C.

The thermograms of the two different naphthenic base oils are seen in **Figures 12a** and **b** on the following page. Both thermograms are quite similar and considerably different from that of the Solvent Neutral. The naphthenic oils show a lower level of heat transmission such that they produce single peaks which crown almost at the target temperature of 480°C. Again, very little deposits are found.

It would be of interest to run the TEOST Protocol 33C on base oils of differing molecular character but similar
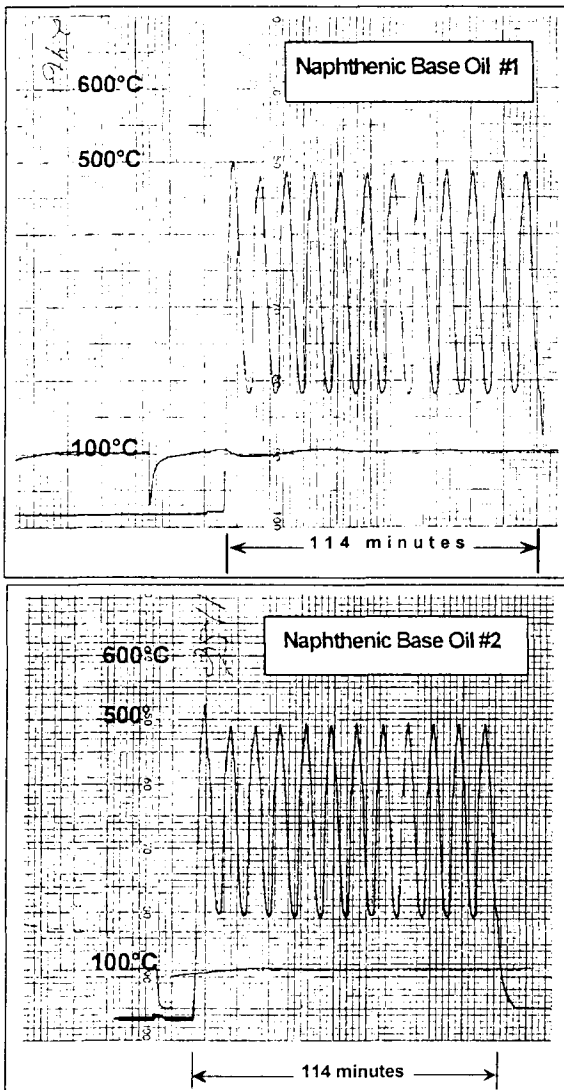
Figure 12a-b: TEOST Protocol 33C thermograms of two naphthenic base stocks. Total weights were 3.6 and 6.2 mg for Naphthenic Base Stock #1 and #2, respectively.

viscosity at the higher temperatures generated by the heating cycles.

TEOST STUDIES OF LOWER TEMPERATURE ENGINE DEPOSIT PROTOCOLS

GENERAL - A more endemic deposit problem has accompanied automobile and heavy duty engines since they were developed. This is the problem of deposits in the engine -- on the piston, on the valves, on the cylinders -- everywhere heat and oxidation produce conditions converting the engine oil into a varnish and/or a carbonaceous mass entrapping contaminants, additives, and their ash.

This more general area of deposit concern has, as a consequence, been a focus of expensive and demanding engine tests for more than 50 years. Over this period, oils have been improved and engine tests have grown more onerous and less repeatable in the effort to distinguish among generally ever-better engine oils.

During this same period of time many laboratory oxidation tests have been developed, each of which had certain applications and supporters. A number of these tests were, at least for some period of time, held proprietary within the laboratories of lubricant manufacturers since such test equipment gave a commercial edge to the quality of their products and the ease of passing expensive engine tests.

As a result of the apparent success of the application of the TEOST apparatus in regard to turbocharger correlation, recent efforts have been directed toward the more challenging area of engine deposits. This portion of the paper is to give a preliminary view of these efforts.

PRELIMINARY TEOST STUDIES - Initial Protocol - The environment of the engine (as opposed to the engine turbocharger) can produce deposit-forming areas having temperatures at least as high as 300°C in the ring-belt area and on the crown lands of the piston. Temperatures in these areas and the gaseous, oxidative nature of the environment produce deposits that are believed to be essentially independent of cyclic conditions.

Accordingly, in these first studies to be reported, a steady-state temperature of 300°C was chosen in the Depositor Rod zone and a temperature of 150°C in the Reactor. Simulated crankcase gases comprising NO_2, SO_2, and CO were permitted to flow into the oil just before the oil entered the Depositor Rod casing. It was found desirable to use diffusers to introduce the gases into the passing oil in a stream of fine bubbles. NO_2 was introduced in one inlet and a combination of SO_2, and CO was introduced into another. These gases were used at levels of 0.5% each of the latter two and 2% of the former with helium as a carrier. Rate of flow was approximately 12 mL/min of NO_2 and 24 mL/min of the combination SO_2, and CO. Thus, the ratio of the three gases was 2:1:1.

A number of protocols were investigated. Variations of run time, flow rate, sample size, and Reactor temperature were among the changes. Three CEC European reference oils used in TU3M engine tests provided the necessary comparators. In one of the more promising protocols, oil circulation rates were the same as in Protocol 33C and the quantity of oil was maintained at 100 mL in the Reactor with an increase of ~20 mL in

Table 3 - New Operating Parameters	
Parameter	Value
Test time	4 hours
Total oil volume	120 mL
Oil pumping rate	0.40 g/min
Reactor temperature	150°C
Depositor temperature	300°C
Moist air flow	3.6 mL/min
NO_2 at 2% conc.	12 mL/min
SO_2 + CO, 0.5% conc. ea.	24 mL/min
Iron naphthenate	100 PPM Fe
Position of controlling thermocouple in Rod	~71 mm from top of Rod

the lines for a total of 120 mL. In all protocols, the procedures

of Protocol 33C were used in determining deposition weights. The list of variables is given in **Table 3**.

Table 4 - European Reference Oil Qualities [10]

Engine Oil	Ring Sticking (10 = all free)	Viscosity Increase	Piston Varnish (100 = clean)	TAN Increase
RL 187/1 (Low)	7.3	65%	31.2	164.8%
RL 193 (Borderline)	9.8	20%	60.8	48.1%
RL 194 (High)	9.9	14%	70.1	42.9%

Reference Oils - The three European engine reference oils used were RL 187/1 (low performance oil), RL 193 (borderline performance oil), and RL 194 (high performance oil). These oils were characterized as shown in **Table 4**.
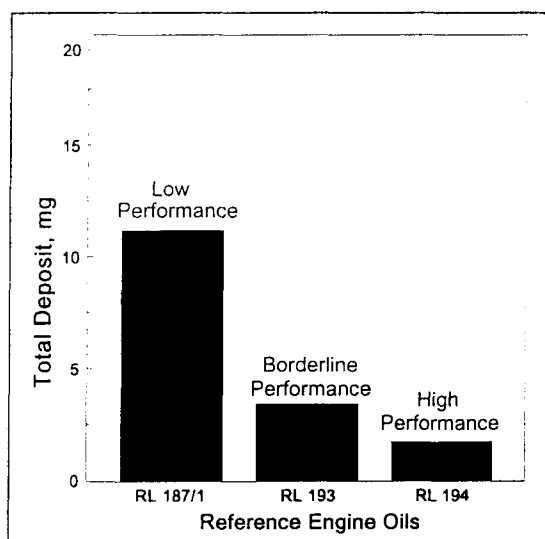
Figure 13: Analyses of three European reference engine oils using experimental TEOST protocol.

Results - Using the protocol given in **Table 3**, the deposit results obtained are shown in **Figure 13**. Not surprisingly for a piston varnish comparator, the deposit levels are relatively low compared to Protocol 33C results on various oils at much higher temperatures. However, the order shown in **Figure 13** conforms reasonably well with the characteristics of these oils given in **Table 4** -- as is also shown in **Figure 14**. That is, the low performing Reference Oil RL 187 shows higher deposits on the Depositor Rod and high performing Reference Oil RL 194 shows lower deposits on the Depositor Rod of the three oils available for study. The fact that considerable data have been collected on these oils in European engine tests has been helpful in directing the authors along productive lines of study.

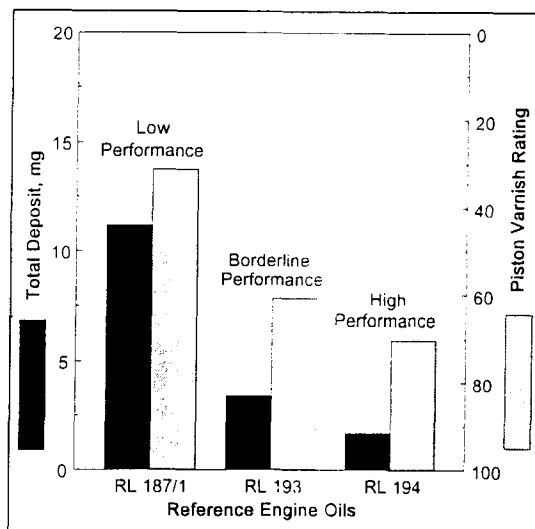**Comparison of Protocol 33C to Initial Lower-Temperature-Protocol Results** - To see how these three

Figure 14: Comparison of results from experimental TEOST protocol with those from engine tests of three European reference engine oils.
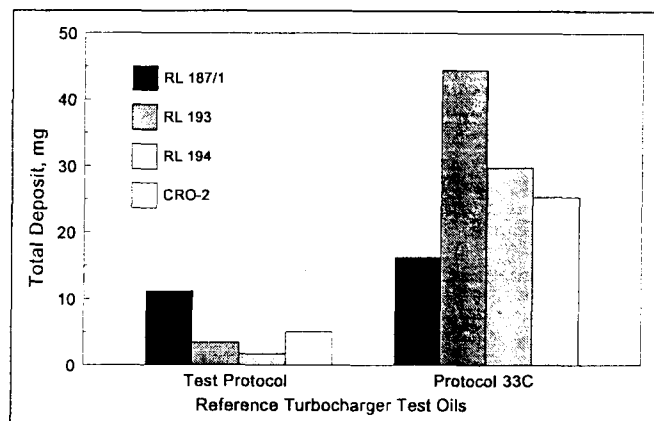
Figure 15: Comparison of results from experimental TEOST protocol on the three European reference engine oils against Protocol 33C.

reference oils would respond to Protocol 33C, the oils were run in this latter mode and the data are plotted in **Figure 15** for not only the above three engine oils but also for the oil used as an acceptable reference engine oil in Protocol 33C -- CRO-2. Interestingly, in regard to the European reference oil series, the order is considerably different, particularly with both the higher and lower deposit levels which are inverted. Indirectly, this information can be interpreted to suggest that the deposit-forming mechanisms in the turbocharger and in the engine are fundamentally different. This may simply be attributed to the difference in level of oxidation-producing chemistry and its impact on deposit-formation.

Results on RL 194 and CRO-2 indicate that for some time it has been possible to blend oils reducing both turbocharger and engine deposits. The CRO-2 is shown to be marginal in comparison to the European reference engine oils -- a reasonable reflection of its designation of API SF. Results with RL 194, in turn, may suggest somewhat greater awareness and focus in Europe to simultaneously protect both the engine and the turbocharger.

DISCUSSION

DEPOSIT FORMATION - The intricate relationship between additive composition, heat transfer, oil and additive response to heat -- either in the presence or relative absence of oxidizing conditions -- encompasses virtually the whole process of deposit formation. In comparison to cyclic studies, typical steady-state deposit studies conducted either in engines or on the bench by observing the amount or rate of deposit can be considered a somewhat limited view of the total process.

Development of TEOST Protocol 33C, was founded on correlation with four turbocharger reference oils. In the process of developing this protocol, cyclic conditions were used. This was in emulation of the conditions producing such deposits in the field where exhaust heat bleed-back down the turbine shaft on shutting off the engine carbonized the less resistive engine oils under conditions leading to thermal decomposition of these oils.

While deposit formation in TEOST Protocol 33C seems to reflect the intricate relationship among the above-mentioned variables as they affect turbocharger deposits, it is shown by another, more recently developed TEOST protocol that the presence of oxidation-producing chemical species can change the order of such deposit-forming tendencies. While this latter protocol is, at this stage, a product of initial studies, it was found to correlate with moderately high temperature, oxidizing conditions generating varnish deposits, viscosity increase, TAN increase, and stuck piston rings.

The data presented seem to suggest that there are at least two primary mechanisms of deposit formation depending on whether oxidizing entities are strongly present or almost absent. Further work with the newer protocols using some of the available techniques of studying the changing character of the test oil exposed to heat and oxidation should be interesting.

HEAT TRANSFER - One of the more critical roles of the engine oil in hot regions of the engine is to carry away heat -- the higher the temperature, the more important the heat transfer function. On the other hand, if the oil is not sufficiently mobile, it becomes vulnerable to the heat it is supposed to remove since that heat and its effects are concentrated on a much smaller volume of engine oil. The consequences of inadequate oil flow in high temperature regions is clearly evidenced in the growth of turbocharger deposits to the point of creating failure of the unit.

<u>Relationship Between Heat Demand and Heat Transfer</u> - A cyclic program such as that of TEOST Protocol 33C imposes a balance among

1. the heat output demanded of the Rod by the program,

2. the heat energy able to be transmitted from the metal to and through the adhering boundary layer at the metal surface to the surrounding oil, and

3. the 'back-reflection' effect of any untransmitted heat on the controlling/sensing thermocouple which responds by reducing the electrical energy to the Rod and, thus, the intensity of the Rod heat output.

Thus, despite the rigorous heating program encoded into the temperature controller and sustained by a powerful Rod-

heating mechanism, the recorded thermogram of the controlling/sensing thermocouple for a given engine oil will approximate, but never match,

1. the programmed protocol, nor necessarily,

2. the recorded thermogram of other oils of different composition.

Thus, to the degree possible within the variables of the process of surface wetting, cleansing, and deposit formation, thermograms of the same oil will give similar appearances. In contrast, engine oils of different additive composition may, and do, show somewhat different thermograms reflecting differences of their immediate and cumulative effects on heat transmissivity.

Moreover, such differences are tempered by the heat transfer characteristics of the base stocks which were indicated to have significant differences depending on their molecular content and, most likely, their chemical and physical properties. Also, the heat transfer characteristics of the finished, formulated engine oils will be affected by the concentration and type of additive systems. To understand the thermograms more completely, the interrelationship of the factors of base stock contribution, additive contribution and additive concentration must be studied.

<u>Deposit Effects on Heat Demand and Heat Transfer</u> - As would be expected, the occurrence and growth of deposits on a metal surface will affect heat transfer to the surrounding oil. Most often, the deposits are shown to further reduce heat transfer from the metal to the oil. In some cases and at certain deposit depths, however, it is found that the deposits will increase the rate of heat transfer in comparison to the early effects of the oil which may have less heat transferring ability. It also must be considered that such increased heat-transferring capability of the deposit may show the influence of the greater surface area presented by the deposited mass to the surrounding and flowing oil.

NEW EXPERIMENTAL PROTOCOL - The work thus far on developing the lower temperature protocol first mentioned as an objective in the earlier paper by the authors [1], seems to be promising. Approximation of engine operating conditions using simulated crankcase gases show reasonably good correlation with European Reference Engine Oils. However, work remains to try to increase the level of these deposits and increase the sensitivity of the test.

At this stage it is evident that the versatility of the TEOST apparatus is valuable when seeking alternative approaches and simulated performance levels for engine oils.

CONCLUSIONS

Through the experiments reported in this paper, the TEOST is shown to be a broadly variable instrument which can be applied to specific problem areas in the field of thermal and oxidative decomposition and deposit. A previous introductory paper showed its application to the problem of engine oil coking in the turbocharger at very high temperatures [1]. This paper continued the presentation of that concept with emphasis on those factors generally not fully understood or appreciated when the method is compared to simpler, steady-state oxidation tests.

DEPOSITS - In particular, TEOST Protocol 33C has been shown to correspond to turbocharger conditions in producing both relative amounts and character of deposits. Further study of the information from the test thermograms indicated the potential value of such data as a reflection of the heat transfer characteristics of

1. the formulated oil,

2. the effects of its additives, and

3. the influence of the deposit mass.

Moreover, x-ray fluorescence spectrograms of the carbonaceous/decomposed-additive mass found in failed turbochargers and those obtained in scrapings from the TEOST Depositor Rods show considerable qualitative similarity. This was believed to indicate that the conditions under which deposits are laid down in the TEOST test are reasonably similar to those formed in the turbocharger over a much longer period of time.

Infrared studies of the engine oil from the Depositor Rod casing in a TEOST test of a borderline high deposit oil shows considerable additive decomposition as well as some oxidation, nitration, and sulfonation. It is believed that these conditions reflect those in the turbocharger, adding further support to the findings from the x-ray fluorescence studies. Future studies of less decomposition-susceptible oils by these infrared techniques should be interesting.

HEAT TRANSMISSION - Another part of the study reported herein was in relation to understanding the information contained in the thermograms of the thermocouple placed in the Rod at the hottest location to sense and control the Rod temperature. Such heat transmission at the Rod-oil interface through any adsorbed additive molecules would be expected to influence both the response of the Rod, its influence on the sensing/controlling thermocouple, and the character and extent of deposition. It was shown that the imposition of cyclic conditions gave information unavailable in steady-state tests concerning an important aspect of lubricating oil -- the ability of the oil to conduct heat away from a hot surface. This same property of heat transmission coupled with dispersant additives is believed directly related to the oil effect on turbocharger deposits and, more broadly, anywhere deposits are laid down.

The paper also has considered the influence of deposition on the Rod surface and the effect this developing deposition has on subsequent heat transmission and Rod temperature.

NEW LOWER TEMPERATURE PROTOCOL - Lastly, the paper presented the results of some initial studies using a new protocol simulating the temperatures and gases in the engine crankcase. The deposit results were compared to data obtained on CEC reference engine oils used in TU3M engine tests and they were shown to be similar in ranking the oils.

When the new protocol and Protocol 33C were compared on the same oil, the differing results suggested that the deposit-forming mechanisms in the engine and the somewhat oxygen-deprived turbocharger are dissimilar. However, it was also shown that some engine oils can and do meet both needs.

ACKNOWLEDGMENTS

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